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Jawaharlal Nehru

“Step Out From the Old to the New”

IS 1488 (1989): Pesticides - 2, 4-D Sodium Salt, Technical
[FAD 1: Pesticides and Pesticides Residue Analysis]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS 1488 : 1989

(Reaffirmed ~~1989~~)

2009

Indian Standard

**PESTICIDES — 2, 4-D SODIUM SALT,
TECHNICAL — SPECIFICATION**

(*Third Revision*)

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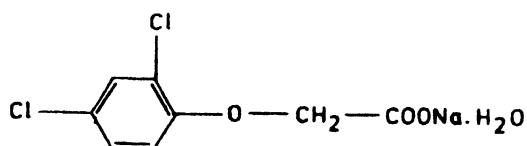
August 1989

Price Group 2

AMENDMENT NO. 1 OCTOBER 1997
TO
IS 1488 : 1989 PESTICIDES — 2, 4-D SODIUM
SALT, TECHNICAL — SPECIFICATION

(*Third Revision*)

(*Second cover page, Foreword, para 4*) — Substitute the following
'Structural Formula' for the existing:



(*Page 1, clause 2*) :

- a) Substitute 'IS 1070 : 1992 Reagent grade water (*third revision*)' for 'IS 1070 : 1977 Specification for water for general laboratory use (*second revision*)'.
- b) Substitute 'IS 10946 : 1996 Method of sampling for technical grade — Pesticides (*first revision*)' for 'IS 10946:1984 Methods of sampling for technical grade pesticides'.

[*Page 1, Table 1, Sl No. (iii), col 5*] — Substitute 'B' for 'A'.

Amend No. 1 to IS 1488 : 1989

[*Page 1, Table 1, Sl No. (v), col 5*] — Substitute 'C' for 'B'.

(*Page 2, clause 6.1*) — Substitute 'IS 10946:1996' for 'IS 10946:1984'.

(*Page 2, clause 7.2*) — Substitute 'IS 1070:1992' for 'IS 1070:1977'.

(*Page 2, clause A-3.2*) — Substitute 'A-2.4' for 'D-2.4', 'A-2.5' for 'D-2.5', 'A-2.2' for 'D-2.2' and 'A-2.3' for 'D-2.3' in lines 1, 5, 11 and 16 respectively.

(*Page 3, clause D-1.1*) — Substitute 'B-2.1' for 'D-2'.

(FAD 1)

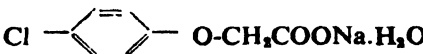
FOREWORD

This Indian Standard (*Third Revision*) was adopted by the Bureau of Indian Standards on 24 April 1989, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council.

The monohydrate sodium salt of 2, 4-dichlorophenoxyacetic acid, namely, 2, 4-D sodium, technical, is extensively used in the control of weeds in agriculture and horticulture.

2, 4-D sodium salt, technical is generally manufactured to contain 80 percent (*m/m*) of 2, 4-dichlorophenoxyacetic acid (2, 4-D).

The empirical and structural formulae and the molecular mass of the product are indicated below:

<i>Empirical Formula</i>	<i>Structural Formula</i>	<i>Molecular Mass</i>
$C_8H_5O_2Cl_2Na.H_2O$		261.0

It the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these rules, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PESTICIDES — 2, 4-D SODIUM SALT, TECHNICAL — SPECIFICATION

(Third Revision)

1 SCOPE

1.1 This standard prescribes the requirements and method of sampling and test for 2, 4-D sodium salt, technical.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title
IS 1070 : 1977	Specification for water for general laboratory use (second revision)
IS 4321 : 1989	Specification for 2, 4-D, technical (second revision)
IS 6940 : 1982	Methods of test for pesticides and their formulations (first revision)
IS 8190 (Part 1) : 1988	Requirements for packing of pesticides : Part 1 Solid pesticides (second revision)
IS 10946 : 1984	Methods of sampling for technical grade pesticides

3 REQUIREMENTS

3.1 **Description** — It shall be light coloured crystalline powder with slight phenolic odour.

3.2 Identity Test

3.2.1 The solubility of the material in 100 ml distilled water at 30 to 32°C shall not be less than 4.0 g.

3.2.2 The *R_f* value of extracted 2, 4-D, when determined by the method prescribed in Annex A shall be same as that of standard 2, 4-D.

3.2.3 The material shall also comply with the requirements specified in Table 1.

4 PACKING

4.1 The material shall be packed in accordance with the requirements given in IS 8190 (Part 1) : 1988.

Table 1 Requirements for 2, 4-D Sodium Salt, Technical
(Clauses 3.2.3 and 7.1)

Sl. No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of this Standard	Annex of IS 4321 : 1989
i)	Acid content, (calculated as 2, 4-dichlorophenoxy acetic acid, 2, 4-D), percent by mass, <i>Min</i>	80	B	—
ii)	Water insoluble matter, percent by mass, <i>Max</i>	0.5	C	—
iii)	Free phenol Content (expressed as 2, 4-dichlorophenol of the declared acid content), percent by mass, <i>Max</i>	1.0	—	A
iv)	Melting point of the extracted acid	136-140°C	D	—
v)	Loss on drying at 105°C, percent by mass, <i>Max</i>	8	—	B

5 MARKING

5.1 The following details shall be marked legibly and indelibly on each container in addition to any other information specified in *Insecticides Act, 1968* and Rules framed thereunder:

- Name of the material;
- Name of the manufacturer;
- Date of manufacture;
- Batch number;
- Acid content, percent (*m/m*);
- Net mass of contents; and
- Minimum cautionary notice as worded in *Insecticides Act, 1968* and Rules framed thereunder.

6 SAMPLING

6.1 Representative samples of the material shall

be drawn as per the methods described in IS 10946 : 1984.

7 TESTS

7.1 Tests shall be carried out as prescribed in the appropriate annexures given in col 4 and 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Clause 3.2.2*)

IDENTITY TEST

A-0 GENERAL

The method involves extraction of 2, 4-D from sample by diethyl ether, concentration and spotting on TLC plates simultaneously with 2, 4-D reference standard and comparison of the two chromatograms.

A-1 APPARATUS

A-1.1 Micropipette

A-1.2 TLC Plates 20 cm × 20 cm and a suitable developing chamber.

A-1.3 Sprayer with Bulb

A-1.4 TLC Applicator, with measuring blades.

A-1.5 Air-Oven

A-1.6 Weighing Bottle

A-2 REAGENTS

A-2.1 Silica Gel G, neutral.

A-2.2 Developing Solvent System Chloroform: acetic acid (95 : 5).

A-2.3 Spraying Reagent, 0.04 percent bromocresol green in alkaline ethanol or methanol (*m/v*).

A-2.4 Sample Solution, 5 percent solution of the extracted residue (*see* D-1) in methanol.

A-2.5 2, 4-D Reference Standard Solution, of known purity; 5 percent (*m/v*) solution in methanol.

A-3 METHOD

A-3.1 Preparation of TLC Plates

Mix about 50 g of neutral silica gel with 85 to 90 ml of water for three minutes to form a uniform slurry. Then by means of the applicator, coat the plate with this slurry to get silica gel layer, of thickness not exceeding 0.25 mm. Activate the plate in the oven maintained at 110°C for 90 minutes.

A-3.2 Procedure

Take 10 μ l of the sample solution (*see* D-2.4) with a micropipette and apply in the form of a uniform spot on the plate at a distance of 3 cm from the bottom edge. Similarly take 10 μ l reference solution of 2,4-D (*see* D-2.5) and apply a spot at a distance of 3 cm from the sample spot on the plate, and at a distance of 3 cm from the bottom. After complete evaporation of the solvent, place the plate in the chromatographic chamber previously saturated with developing solvent (*see* D-2.2) and allow the developing solvent to run up at room temperature to a distance of 10 cm from the point of application of the spot. Take out the plate and dry at 100°C in oven for 20 minutes to remove acetic acid. Then spray with the spraying reagent (*see* D-2.3). The principal spot in the chromatogram obtained with sample solution should correspond at the same *R_f* value to that in the chromatogram obtained with reference standard solution of 2,4-D acid.

ANNEX B

[*Table 1, Item (i)*]

DETERMINATION OF ACID CONTENT

B-1 REAGENTS

B-1.1 Dilute Hydrochloric Acid 1 : 1 by volume.

B-1.2 Diethyl Ether

B-1.3 Ethyl Alcohol Neutral, or alternatively methyl alcohol, neutral may be used.

B-1.4 Bromothymol Blue Indicator Solution 0.04 percent solution in alcohol (*m/v*).

B-1.5 Standard Sodium Hydroxide Solution 0.1 N.

B-2 PROCEDURE

B-2.1 Weigh accurately about 10 g of sample and

transfer it to a 250 ml beaker. Dissolve the sample in about 200 ml hot distilled water at 90 to 95°C temperature. Transfer it quantitatively to a 250-ml volumetric flask, cool to room temperature and dilute up to mark with distilled water. Mix well and filter through a Whatman No. 1 or equivalent filter paper into an Erlenmeyer flask. Discard the first 50 ml of the filtrate and collect the rest of it. Take out with pipette, a 25 ml aliquot from the filtrate into a 100 ml separating funnel. Acidify the solution with about 1.5 ml of hydrochloric acid. Confirm complete splitting of the sample by adding little more hydrochloric acid to the aqueous layer. If white precipitate of 2, 4-D does not form, it confirms complete splitting. Saturate the contents with about 5 g of sodium chloride or saturated sodium chloride solution. Extract the aqueous phase thus saturated with sodium chloride with three successive 25 ml portions of diethyl ether. Transfer the second and third ether extracts quantitatively to the first extractives (total about 75 ml). Wash the combined ether extracts with 10 ml portions of distilled water until the ether solution is free of chlorides (usually 3 to 5 washings). Remove the ether by evaporation on a hot water bath and transfer quantitatively the

residues to a 250 ml Erlenmeyer flask with the help of 60 ml alcohol. Titrate with standard sodium hydroxide solution using bromothymol blue indicator to a green coloured end point. Carry out a blank determination on equal quantity of alcohol and ether used in the test.

B-3 CALCULATION

B-3.1 Acid (2, 4-D) content, percent by mass

$$= \frac{(A-B) \times N \times 221}{M}$$

where

A = volume, in ml, of the standard sodium hydroxide required for the test;

B = volume, in ml, of the standard sodium hydroxide required for blank determination;

N = normality of the standard sodium hydroxide used; and

M = mass, in g, of the material taken for the test.

ANNEX C

[Table 1, Item (ii)]

DETERMINATION OF WATER INSOLUBLE MATTER

C-1 PROCEDURE

C-1.1 Dry a sintered glass or tared Gooch crucible to constant mass at 150°C. Weigh accurately about 10 g of the sample and dissolve in about 400 ml of hot distilled water. Heat to boiling and stir until all water soluble material has dissolved. Filter hot through the prepared crucible and wash three times with hot distilled water. Dry to constant mass at 105°C, cool and then weigh.

C-2 CALCULATION

C-2.1 Water insoluble matter, percent by mass

$$= \frac{m \times 100}{M}$$

where

m = mass, in g, of the residue obtained after drying; and

M = mass, in g, of the material taken for the test.

ANNEX D

[Table 1, Item (iv)]

DETERMINATION OF MELTING POINT OF EXTRACTED ACID

D-0 GENERAL

D-0.1 2, 4-D is extracted from 2, 4-D sodium salt, technical and melting point is determined of the extracted and dried 2, 4-D.

D-1 PROCEDURE

D-1.1 Extract with ether 2, 4-D acid following the

procedure described in D-2 and wash the combined ether extracts with portions of distilled water until the ether solution is free from chlorides (usually 3 to 5 washings are required). Distil off the ether. Dry the flask with the residue at 105±2°C for about 30 minutes. Determine the melting point of the finely powdered residue (which is the extracted 2, 4-D acid) according to the method prescribed in 6 of IS 6940 : 1982.

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